METHOD FOR PREPARING CLEAR POLYORGANOSILOXANE ELASTOMERS

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to polyorganosiloxane elastomers. More particularly this application relates to a method for preparing optically clear polyorganosiloxane elastomers.

2. Description of the Prior Art

Polyorganosiloxane elastomers, which are also referred to in the art as silicone rubbers, exhibit many desirable properties that make these materials suitable 15 for a variety of end use applications. The elastomers are prepared by curing compositions ranging in consistency from pourable liquids to gums. Liquid or pumpable compositions are particularly desirable because they can be fabricated by injection molding.

Some end use applications of polyorganosiloxane elastomers require that the cured elastomer contain a reinforcing filler such as silica, yet be optically clear. Unfilled polyorganosiloxane elastomers are transparent and substantially free of haze, however the physical 25 properties of the cured materials are relatively poor.

When a reinforcing filler such as finely divided silica is added to a curable polyorganosiloxane composition the filler is usually treated with a low molecular weight hydroxyl-containing silane, siloxane or a hexaor- 30 ganodisilazane to prevent a phenomenon known as crepe aging or structuring. Irrespective of the method used to treat the filler, its presence may decrease the not considered optically clear.

Methods for preparing optically clear polyorganosiloxane elastomers containing a treated reinforcing filler are disclosed in the prior art. One of these methods involves using a filler of sufficiently small particle size that light rays passing through the cured elastomer are not refracted even when the refractive indices of the cured organosiloxane reactants do not match those of the silica filler. Methods for preparing 45 silica fillers within this particle size range are described in U.S. Pat. No. 4,418,165, which issued to Polmanteer and Chapman on Nov. 29, 1983 and in U.S. Pat. No. 2.786,042, which issued to Iler on Mar. 19. 1957.

Optically clear cured silicone elastomers can be obtained using commercially available grades of fume silica or other finely divided silicas that are treated with anti-crepe agents if the refractive indices of the durable polyorganosiloxane(s) and the treated filler are substantially equal. This can be accomplished by either select- 55 ing polyorganosiloxane reactants to yield a cured elastomer of substantially the same refractive index as the filler or by treating the silica or other filler to alter its refractive index to match that of the cured elastomer.

For example, it has been found that the refractive 60 indices of organosiloxane copolymers containing dimethylsiloxane and phenylmethylsiloxane units are essentially the same as the refractive index of silica. Other silicon bonded hydrocarbon radicals such as trifluoropropyl can be included in the polymer if adjustments are 65 made to match the refractive indices of the base polymer and the filler. This approach to obtaining optically clear elastomers is less than desirable because it limits

the types of polyorganosiloxanes that can be included in a curable composition.

As an alternative to modifying the structure of the polyorganosiloxane, one can use specified classes of silica treating agents such as are disclosed in U.S. Pat. No. 3.996.189 which issued to Travnicek on Dec. 7, 1976, and in U.S. Pat. No. 3.624,023, which issued to Hartlage on Nov. 30, 1971.

In U.S. Pat. No. 4,008,198, which issued on Feb. 15, 10 1977, Krohberger et al. teach preparing highly transparent or optically clear polyorganosiloxane elastomers by blending together viscous polydiorganosiloxane with (1) at least 0.02 percent by weight, based on the polydiorganosiloxane, of a nitrogen compound having at least one triorganosilyl group wherein the silicon atom is bonded to nitrogen directly or through an oxygen atom and no more than one triorganosilyl group per molecule is bonded directly to nitrogen, (2) from 4 to 40 percent by weight, based on the polydiorganosiloxane, of a hexaorganodisilazane, and (3) from 10 to 150 percent by weight. based on the polydiorganosiloxane, of a silicon dioxide having a surface area of at least 50 m²/gram.

The examples of this patent report that an elastomer prepared by curing a polydimethylsiloxane containing 0.07 mole % of methylvinylsiloxane units exhibits a light transparency value of 91%. The transparency value is reduced to 84% when a hydroxyl terminated polydimethylsiloxane is substituted for hexamethyldisilazane.

By comparison, a composition prepared using a silazane treated filler and a polydimethylsiloxane containing 5.5 mole percent of diphenylsiloxane units and about 0.5 mole % of methylvinylsiloxane units exhibits a light the cured elastomer to the extent that the elastomer is 35 transparency value of 96%. This is consistent with the matching the refractive indices of the filler and the polydiorganosiloxane. No haze values are reported for any of the exemplified compositions.

> Krohberger et al. require both the aforementioned nitrogen compound containing at least one triorganosilyl group and a hexaorganodisilazane to achieve both dimensional stability and optical transparency in the uncured composition. There are no data to indicate whether both compounds are required to achieve a high level of optical transparency.

> The compositions disclosed in the aforementioned Krohberger et al. patent are prepared by mixing the polydiorganosiloxane, silica treating agents and water in a kneader. After an unspecified time period the silica is added. Mixing is continued until the composition becomes homogeneous, at which time the composition is heated at a temperature of 150° C under a pressure of 1 mm. of mercury until there is no evidence of nitrogen compounds being evolved.

> The present inventor was unable to prepare optically clear cured silicone elastomers by following the preparative method exemplified by Krohberger et al. using compositions containing a polydimethylsiloxane as the curable polymer in combination with fume silica and hexamethyldisilazane as the silica treating agent within the concentration range taught by Krohberger et al. The haze value of the cured elastomer was over 40%.

> An objective of the present invention is to provide a method for preparing optically clear cured elastomers from compositions containing a polydimethylsiloxane as the curable polymer and a treated reinforcing silica filler wherein at least a portion of the silica treating